3D EBSD: Tomographic orientation microscopy in a FIB SEM

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Overview

- **Motivation and Features**
- **Methods**

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  Data analysis (GND; interface textures;

  simulation: CPFEM, phase field, MC, CA)
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  DP steels: GND

  ECAP processing: interface textures
- **Conclusions**

Zaefferer et al., Met. Mater. Trans. 39A (2008) 374

Special 3D features of FIB-EBSD tomography

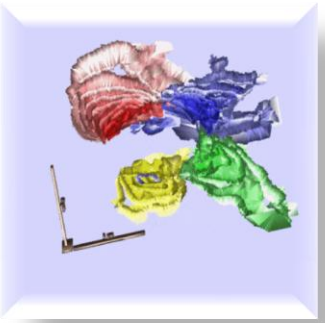
- Increase phase space of microstructure analysis

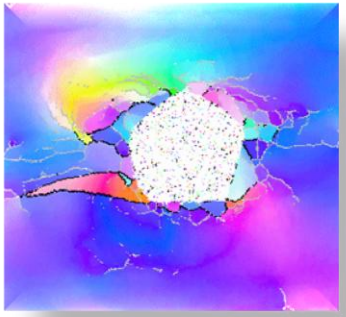
  6D (φ_1,ϕ,φ_2,x,y,z): Crystallography and texture with morphology

  8D (φ_1,ϕ,φ_2,h,k,x,y,z): Interface crystallography (interface texture)

- Spatial texture and phase correlations (connectivity, percolation, correlations)

- Connection to 3D microstructure modeling



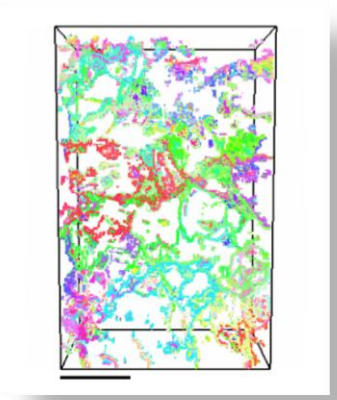


Recent reviews:

- *Uchic et al., MRS Bulletin 32 (2007) 408*
- *Zaefferer et al., Met. Mater. Trans. 39A, (2008) 374*

Advantages of FIB-EBSD tomography (3D EBSD)

- Sectioning by FIB
  - accurate depth definition
  - flat and parallel sections (< 1° deviation)
  - high resolution (< 50 nm)
- Observation by EBSD
  - well-defined contrast on crystalline material
  - ideal for reconstruction of grains in 2D and 3D
  - quantitative description of microstructure
  - high resolution (~ 50 nm)
- Combination of FIB and EBSD
  - high measurement speed
  - fully automatic



Bastos et al.: J. of Microscopy 230 (2008) 487

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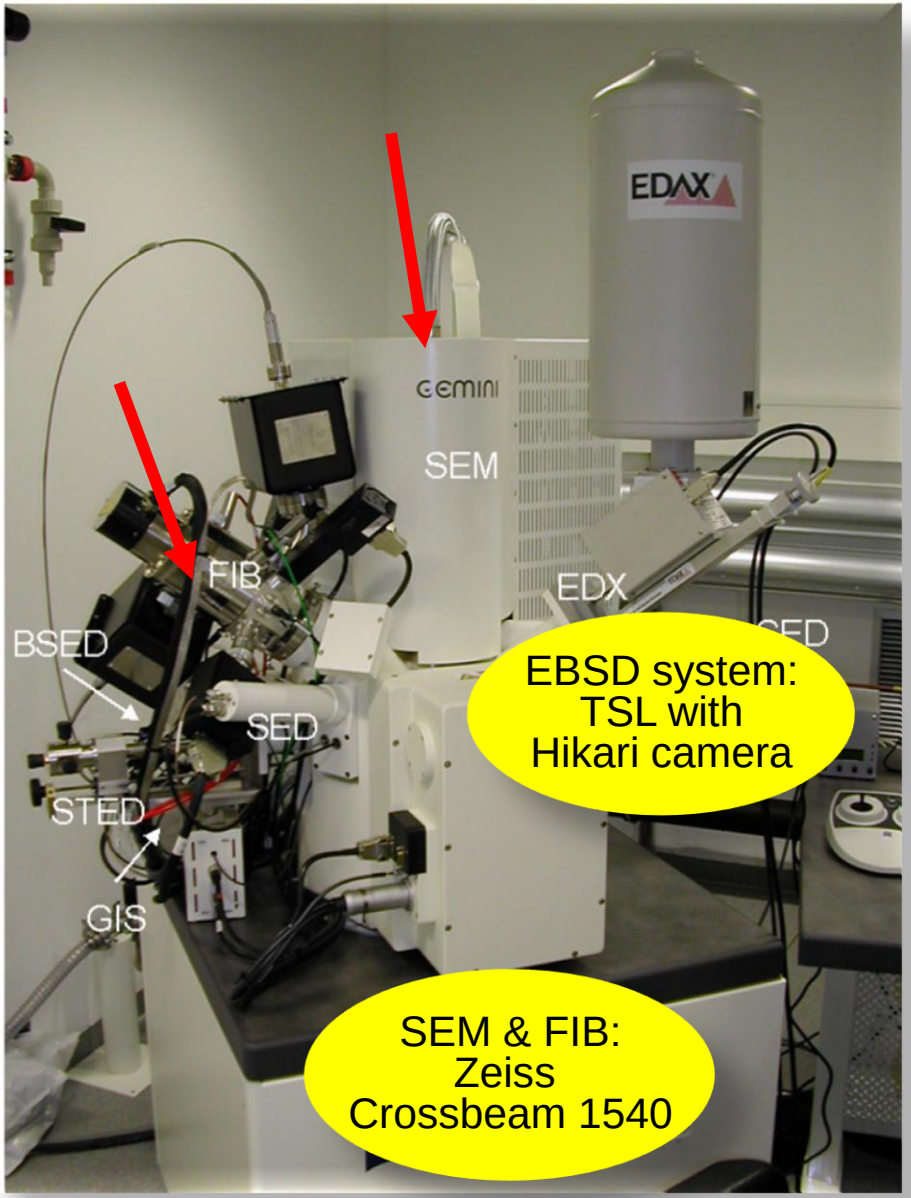
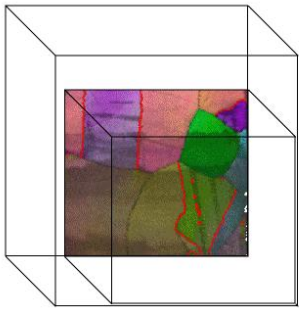
Roters et al. Acta Mater.58 (2010)

Instrument overview

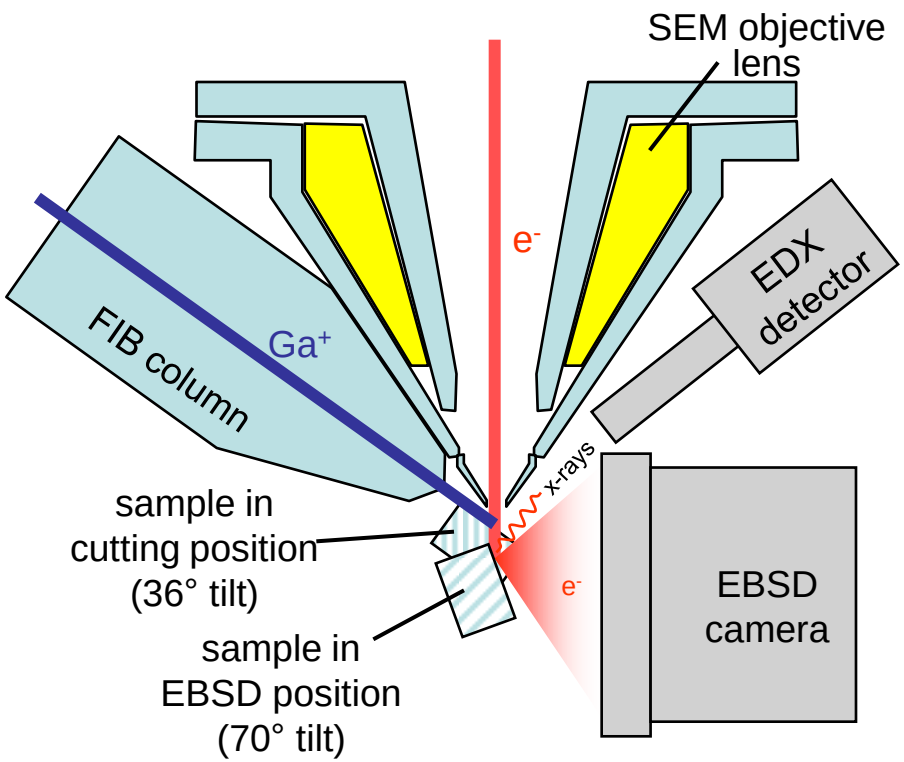


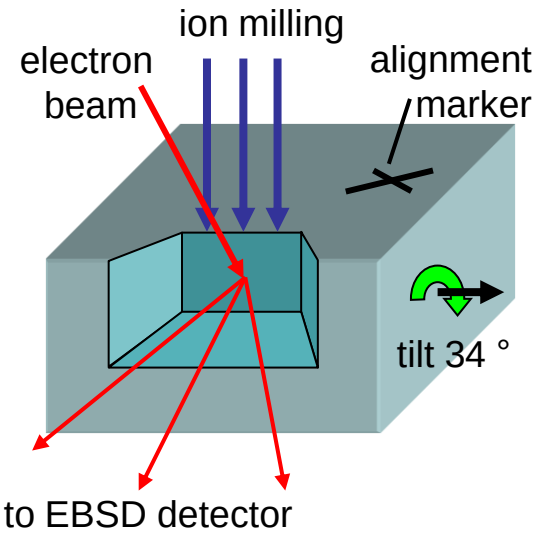
- Scanning electron microscope (SEM)
  - observation of microstructure
- Scanning Ga^+-ion microscope (FIB = focused ion beam)
  - sputtering of material for serial sectioning
- Quantitative images with EBSD and EDX
  - quantitative characterisation of microstructure

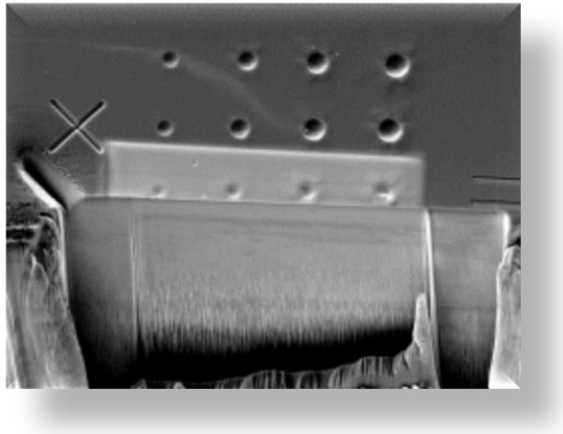


Zaefferer et al., Met. Mater. Trans. 39A (2008) 374

Principle of serial sectioning & orientation microscopy



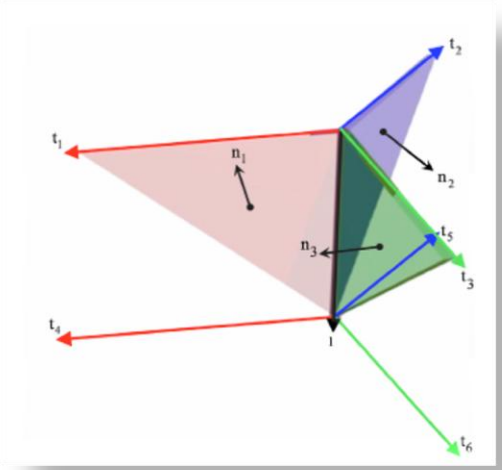




Konrad et al. Acta Mater. 54 (2006) 1369

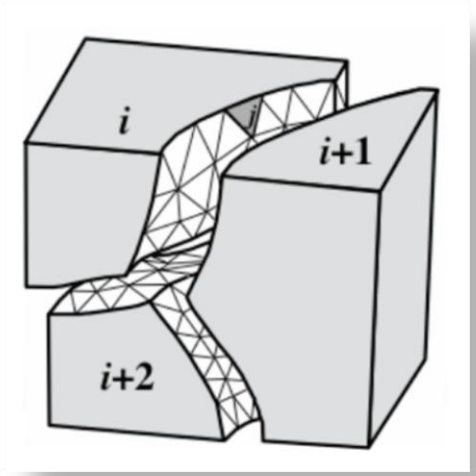
Approaches to 8D interface analysis and 3D GND analysis

Plane boundaries defined by triple junctions in two adjacent layers



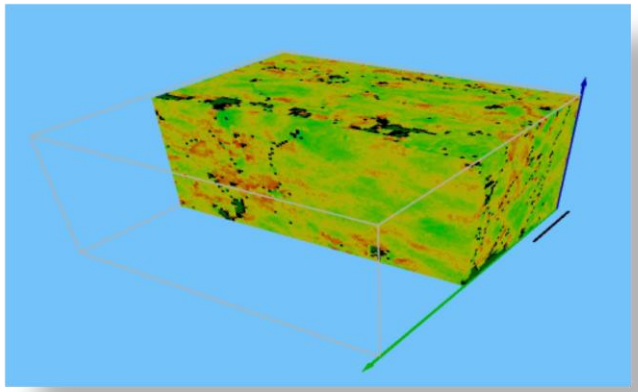
Greg Rohrer

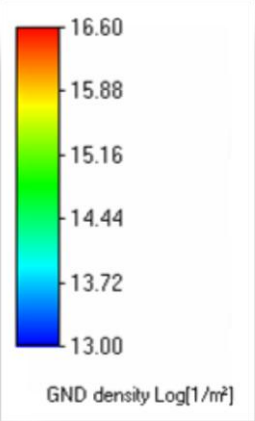
Triangulation the interfaces for defining the grain boundaries



Tony Rollett

GND (Kröner-Nye)





Demir, Raabe, Zaafarani, Zaefferer: Acta Mater. 57 (2009) 559

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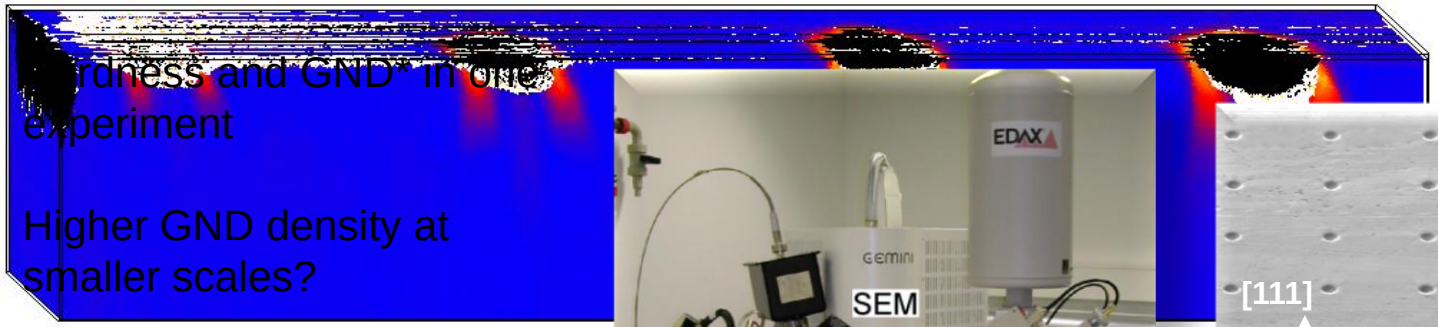
Zaefferer et al., Met. Mater. Trans. 39A (2008) 374

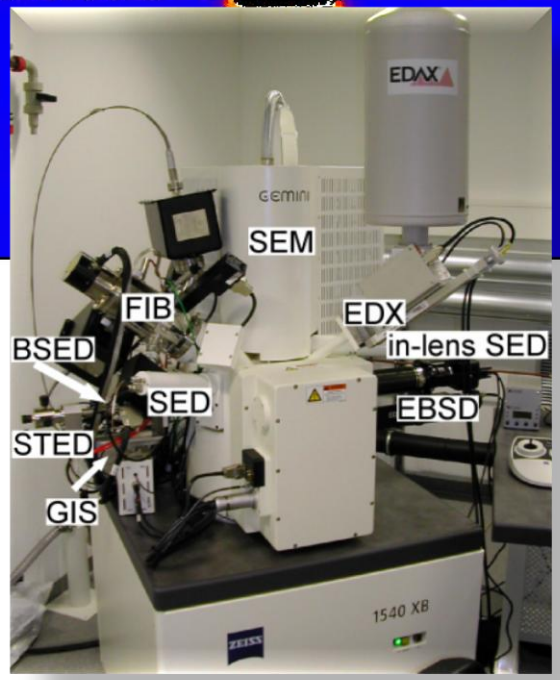
Nanoindentation (smaller is stronger): 3D EBSD and CPFEM

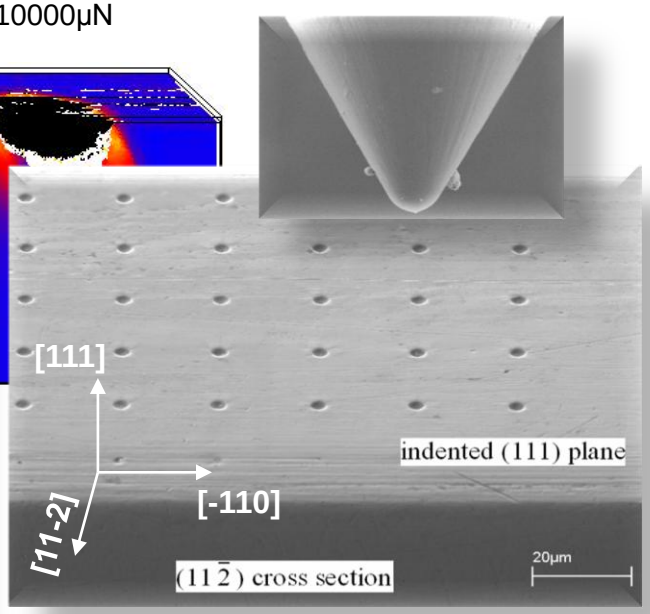
Cu, 60° conical, tip radius 1µm, loading rate 1.82mN/s, loads: 4000µN, 6000µN, 8000µN, 10000µN



Hardness and GND* in one experiment

Higher GND density at smaller scales?





** GND: geometrically necessary dislocations (accomodate curvature)*

Zaafarani et al. Acta Mat. 54 (2006) 1707; Wang et al. Acta Mat. 52 (2004) 2229

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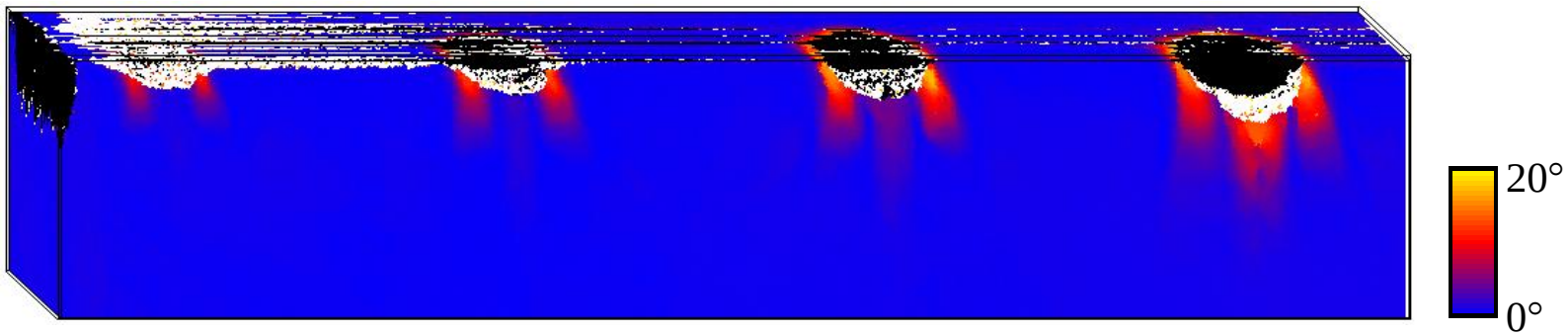

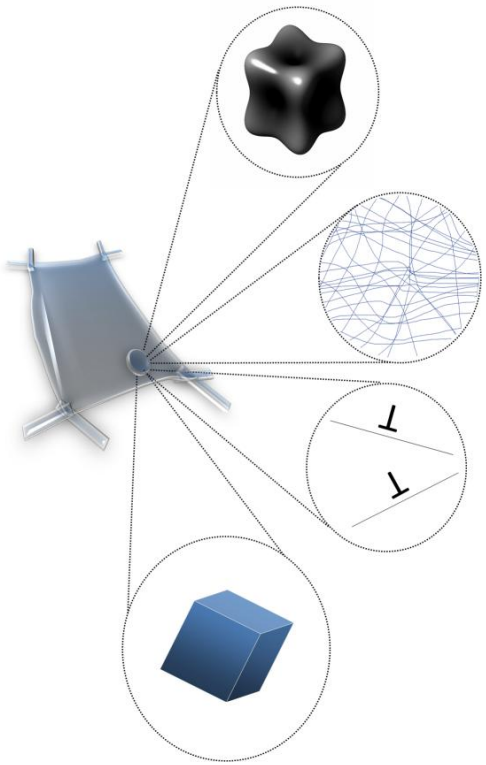

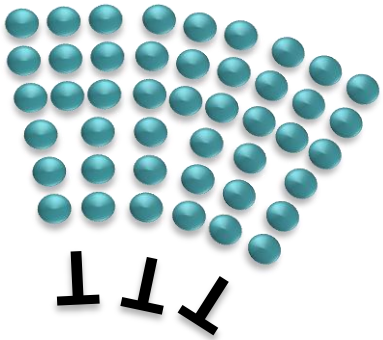

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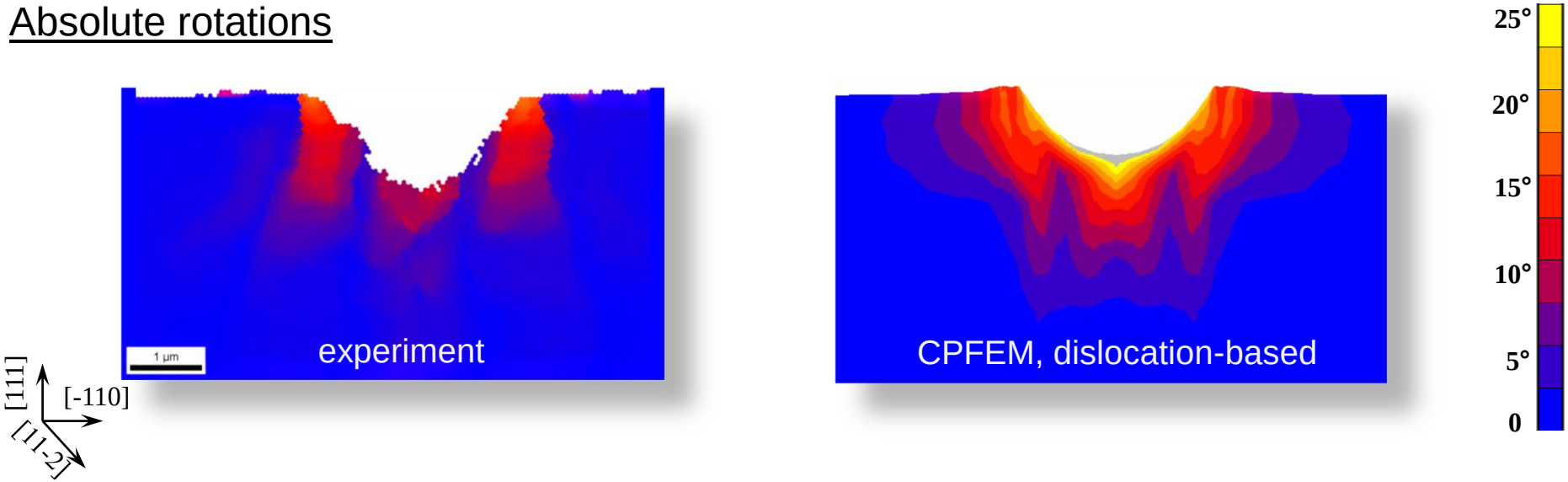
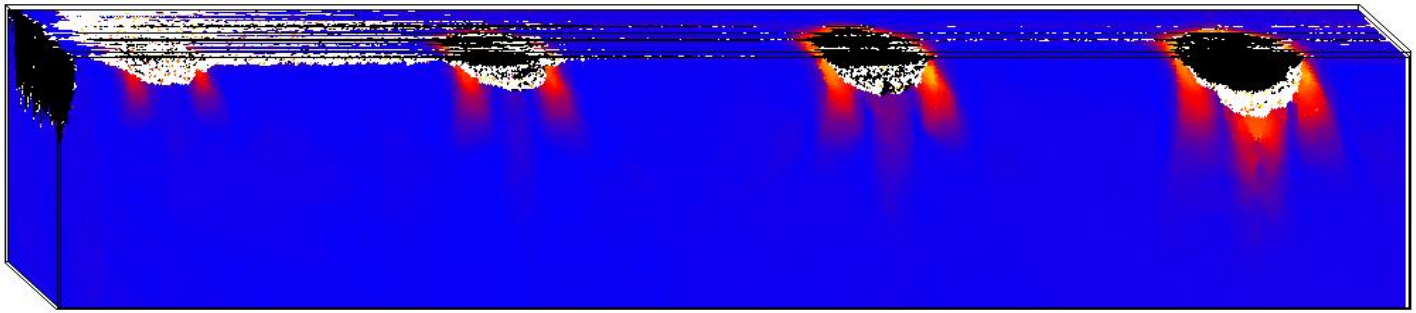
Comparison, crystal rotations (absolute, about [11-2] axis)

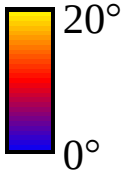
Absolute rotations



Zaafarani, Raabe, Roters, Zaefferer: Acta Mater. 56 (2008) 31

From local misorientations to GNDs



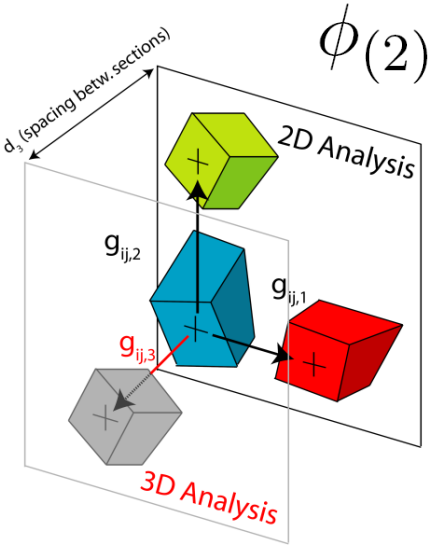


misorientation angle

$$\triangle\phi = \phi_{(2)}\ \phi_{(1)}^{-1}$$ misorientation

$$|\triangle\phi| = min\{cos^{-1}\{tr[(O_i^{cry}\phi_{(1)})\phi_{(2)}^T O_j^{cry}]\}\} \quad i = 1\ldots24,\ j = 1\ldots24$$

$$\phi_{(2)} - \phi_{(1)} = (\triangle\phi - I)\phi_{(1)}$$ orientation difference



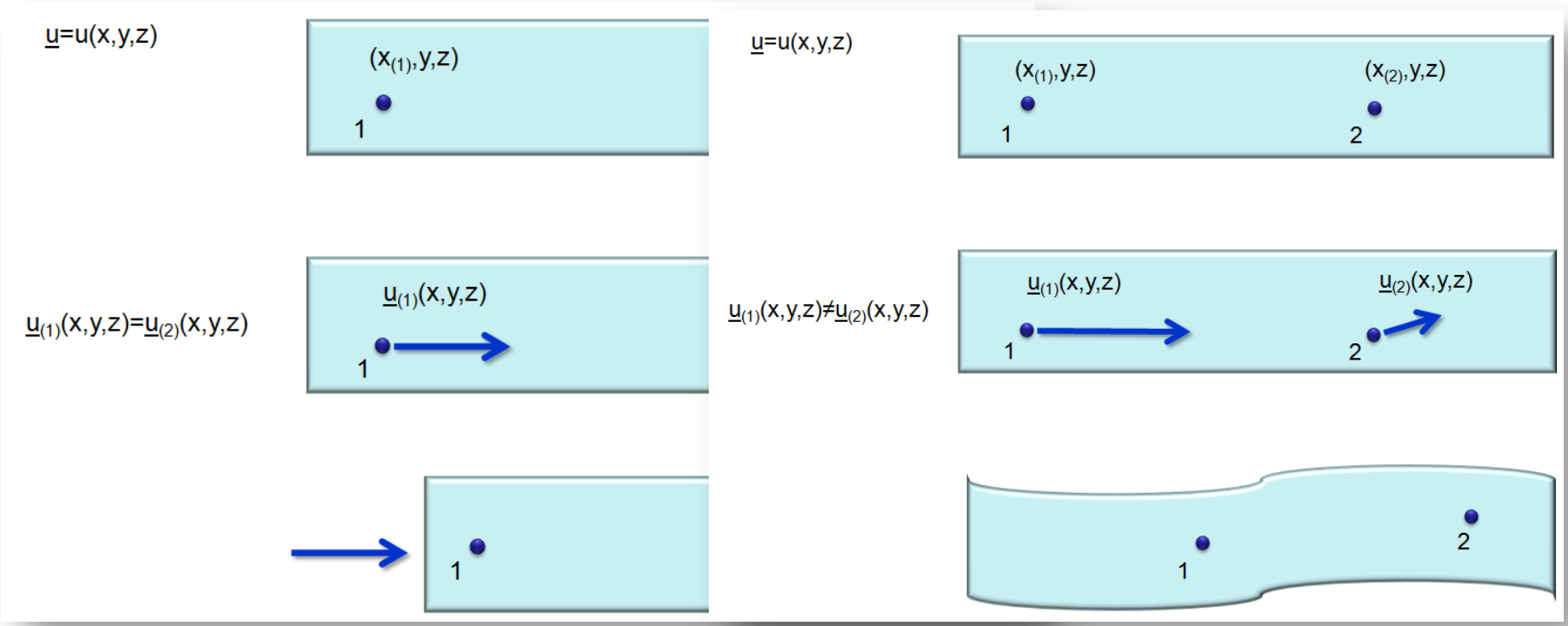
$$g_{ij,k} = \frac{\phi_{(2)ij} - \phi_{(1)ij}}{d_k}$$

orientation gradient
(spacing d from EBSD scan)

From local misorientations to GNDs

$$\beta_{ij} = \frac{\delta u_i}{\delta x_j} = \beta_{ij}^{el} + \beta_{ij}^{pl}$$

distortion
(sym, a-sym)



From local misorientations to GNDs

$$\beta_{ij} = \frac{\delta u_i}{\delta x_j} = \beta_{ij}^{el} + \beta_{ij}^{pl}$$

distortion
(sym, a-sym)

$$\alpha = \nabla \times \beta^{el}$$

$$\alpha_{pi} = e_{pkj} \left(\epsilon_{ij,k}^{el} + g_{ij,k} \right)$$

$$\alpha_{pi} = e_{pkj} \; g_{ij,k}$$

dislocation tensor (GND)

J. F. Nye. Some geometrical relations in dislocated crystals. Acta Metall. 1:153, 1953.
E. Kröner. Kontinuumstheorie der Versetzungen und Eigenspannungen (in German). Springer, Berlin, 1958.
E. Kröner. Physics of defects, chapter Continuum theory of defects, p.217. North-Holland Publishing, Amsterdam, Netherlands, 1981.

From local misorientations to GNDs

Slip and line directions of dislocations for GNDs in a FCC crystal

$\sqrt{2}\,\hat{\mathbf{b}}$:	$\bar{1}10$	$10\bar{1}$	$0\bar{1}1$	$\bar{1}\bar{1}0$	101	$01\bar{1}$	110	$\bar{1}01$	$0\bar{1}\bar{1}$	$1\bar{1}0$	$\bar{1}0\bar{1}$	011	110	101	011	$\bar{1}10$	$10\bar{1}$	$0\bar{1}1$
$\sqrt{6}\,\hat{\mathbf{t}}$:	$\bar{1}\bar{1}2$	$\bar{1}2\bar{1}$	$2\bar{1}\bar{1}$	$\bar{1}1\bar{2}$	$\bar{1}\bar{2}1$	211	$1\bar{1}\bar{2}$	121	$\bar{2}\bar{1}1$	112	$1\bar{2}\bar{1}$	$\bar{2}1\bar{1}$	110	101	011	$\bar{1}10$	$10\bar{1}$	$0\bar{1}1$

$$\mathbf{B} = \mathbf{b}(\hat{\mathbf{t}} \cdot \mathbf{r}) = (\mathbf{b} \otimes \hat{\mathbf{t}})\mathbf{r}$$

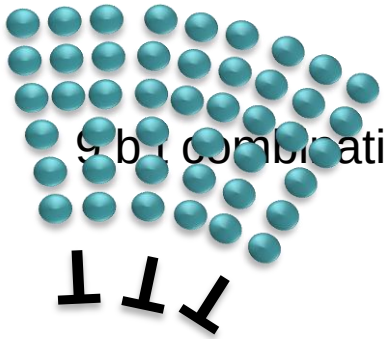
Frank loop thro

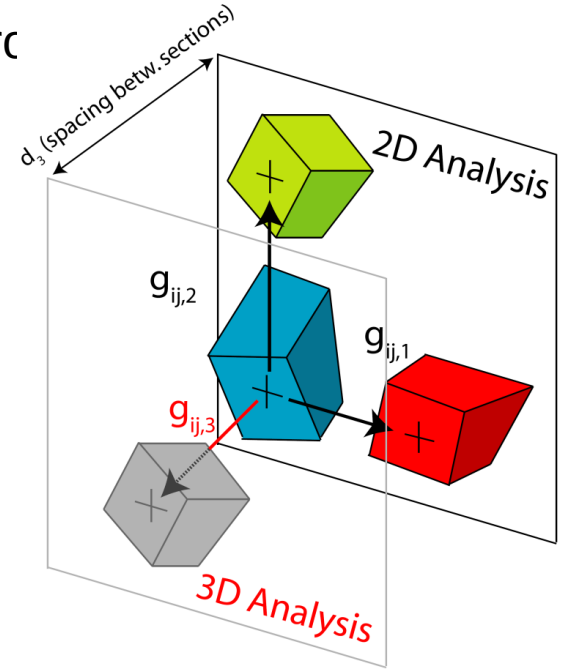
$$\alpha_{ij} = \sum_{a=1}^{18} \rho_{gnd}^{a}\, b_i^a t_j^a$$

18 b,t combination

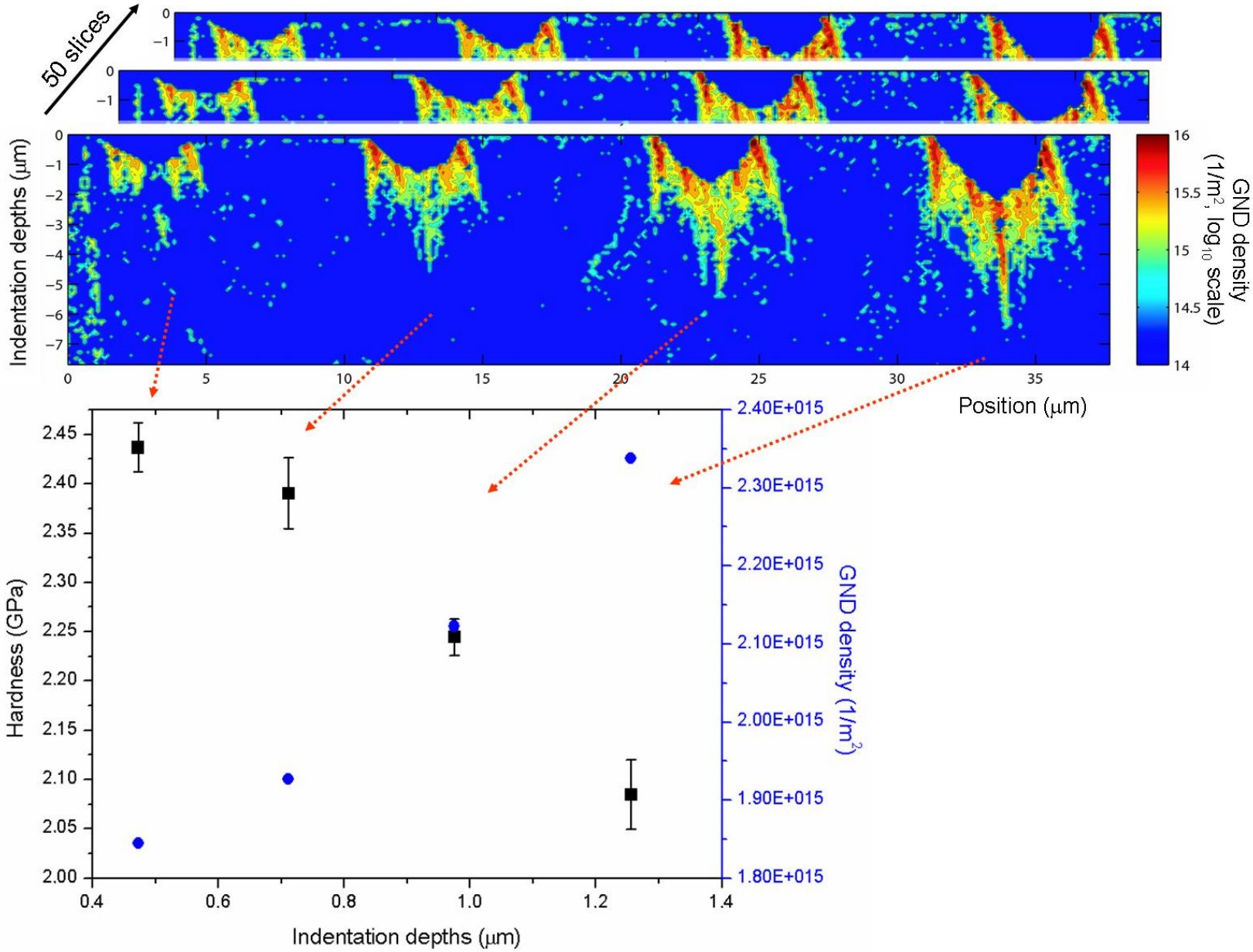
$$\alpha_{ij} = \sum_{a=1}^{9} \rho_{gnd}^{a}\, b_i^a t_j^a$$

9 b,t combinations





Extract geometrically necessary dislocations



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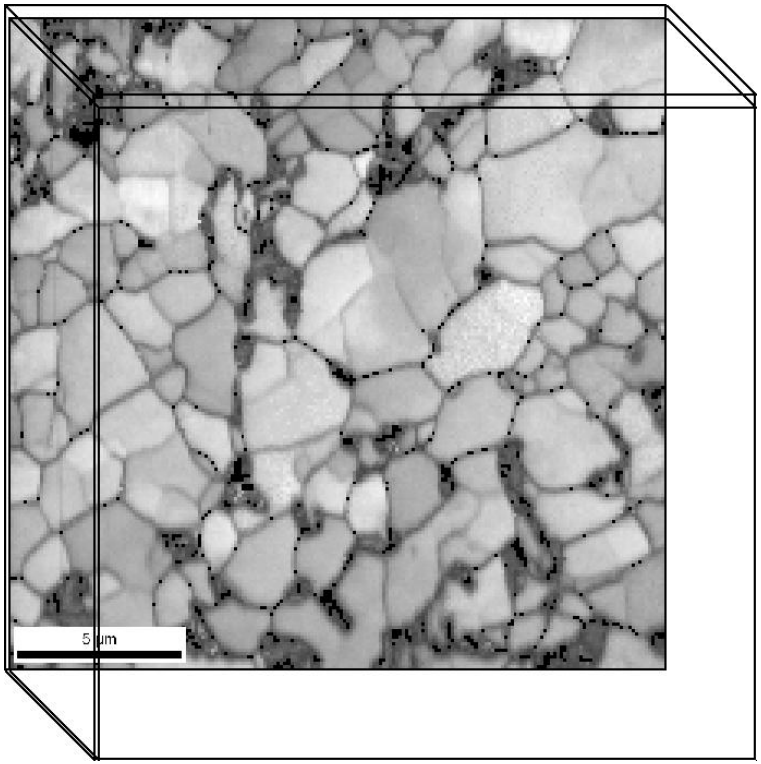
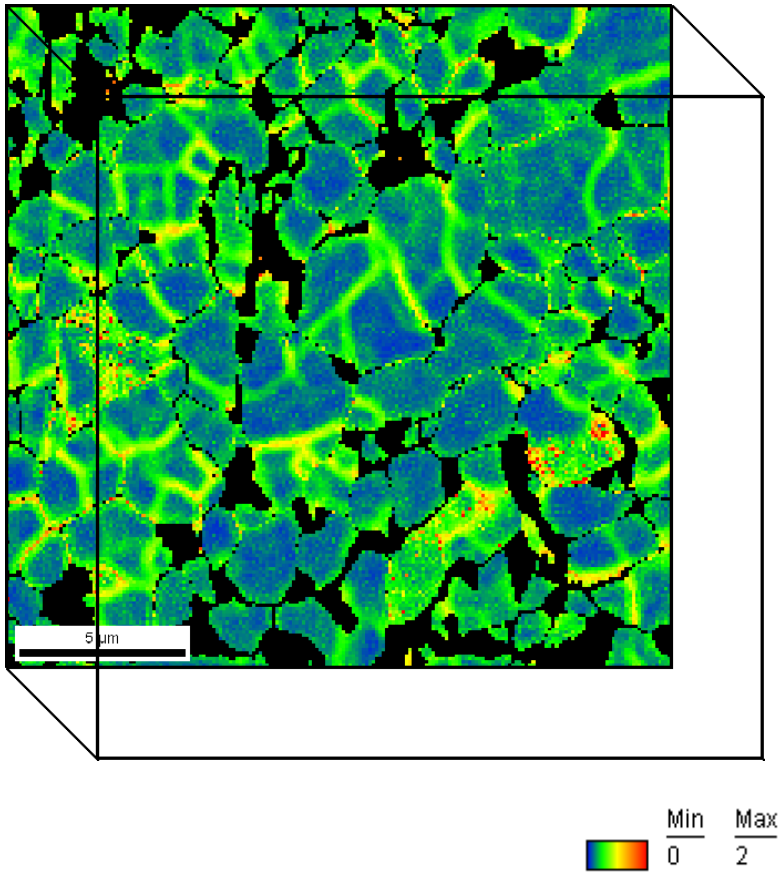
Zaefferer et al., Met. Mater. Trans. 39A (2008) 374

3D-EBSD - results

Image Quality

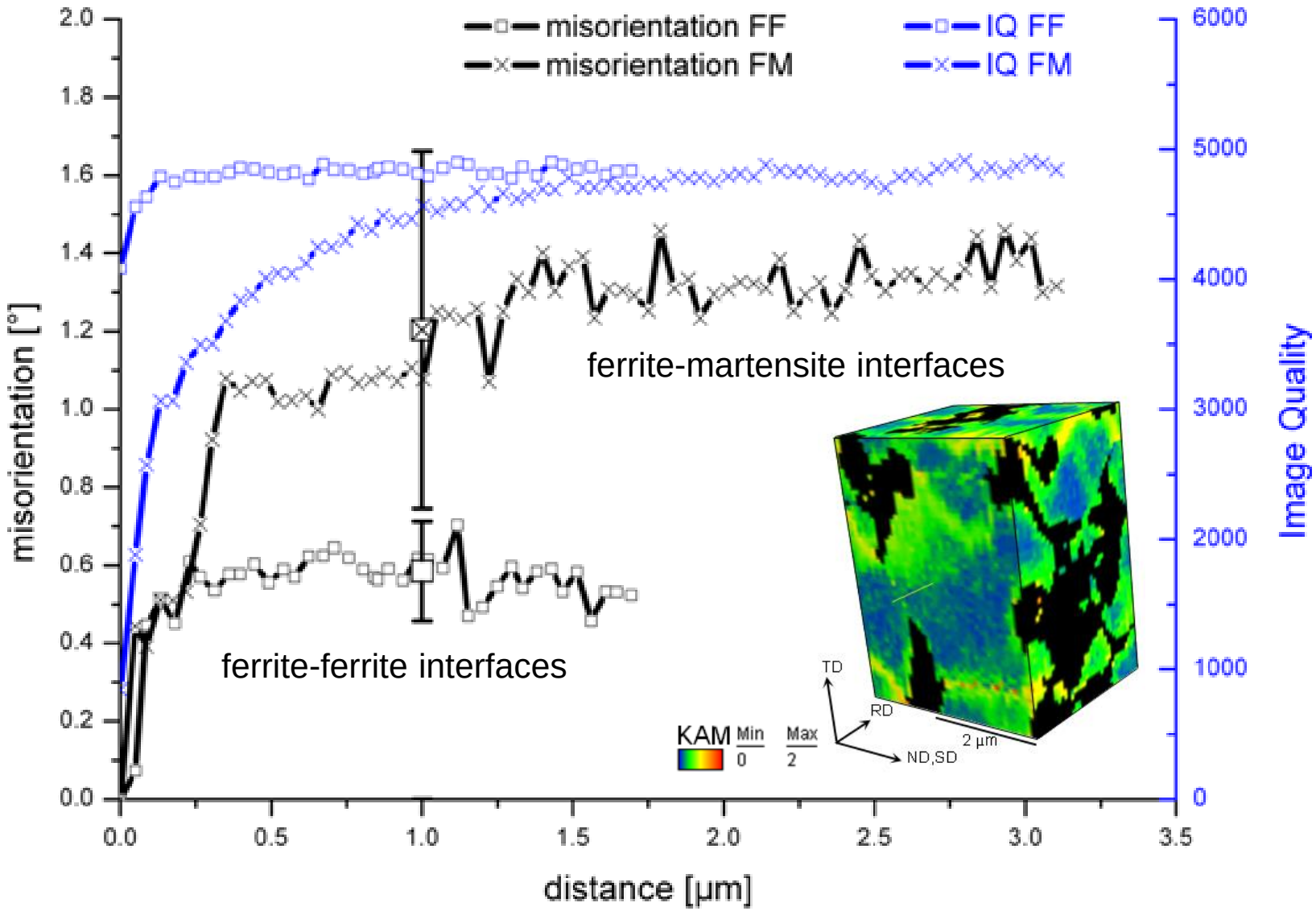


Kernel Average Misorientation
(martensite highlighted in black)



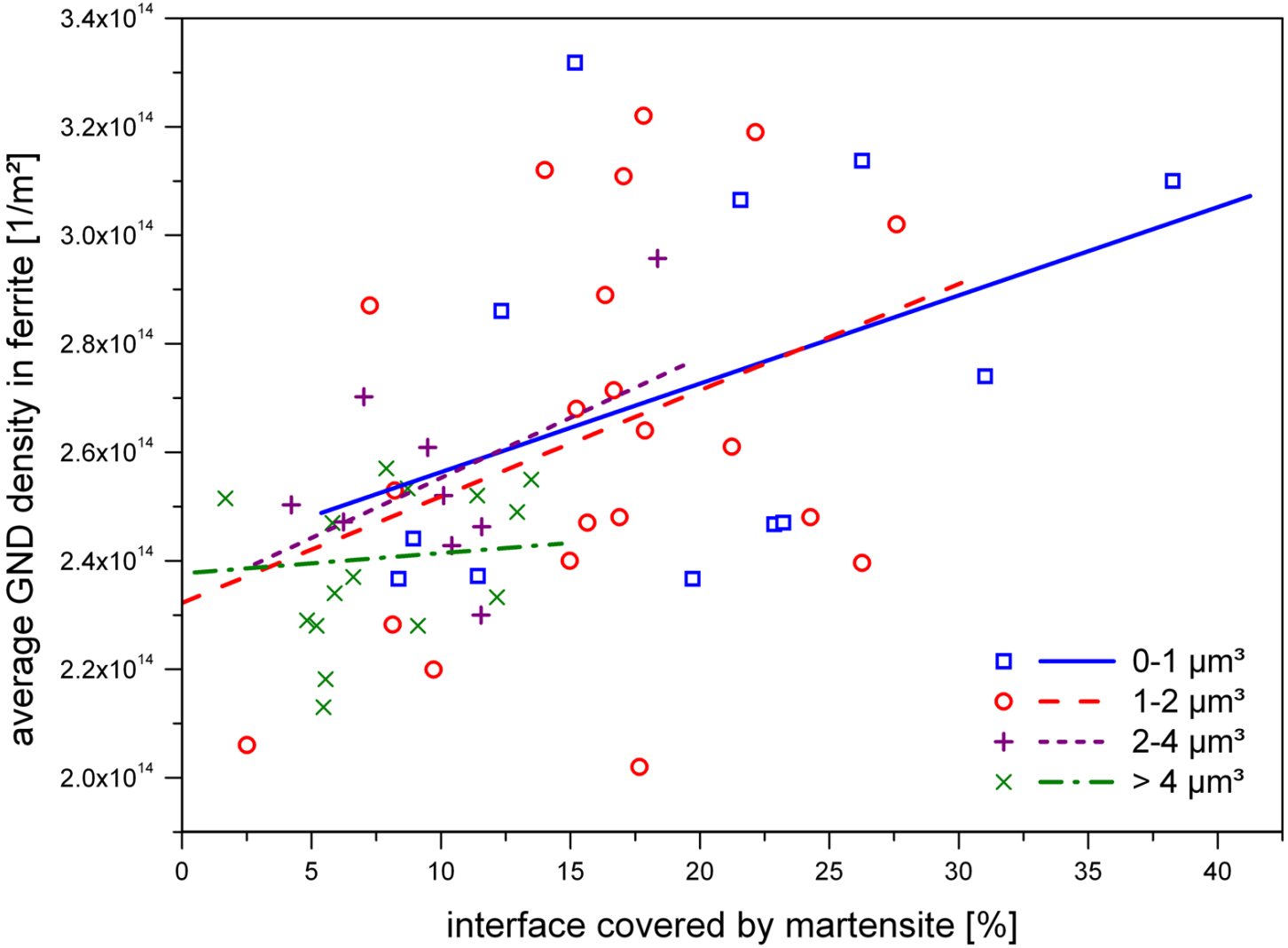
Calcagnotto et al. ISIJ 48 (2008) 1096

DP steel micromechanics

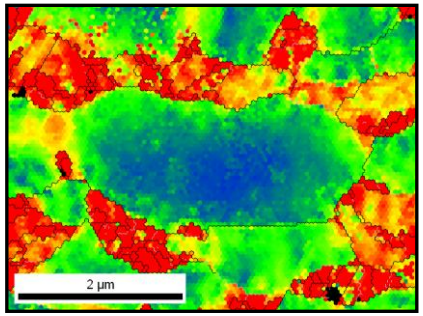


Calcagnotto et al. Mater. Sc. Engin. A 527 (2010) 2738

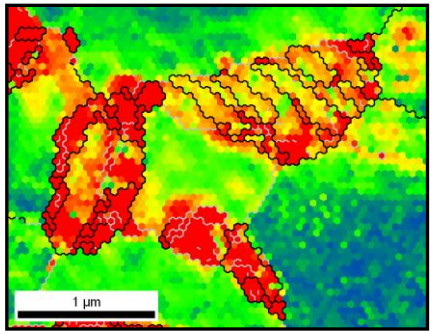
GND density vs. FM interface fraction



large grain



small grain



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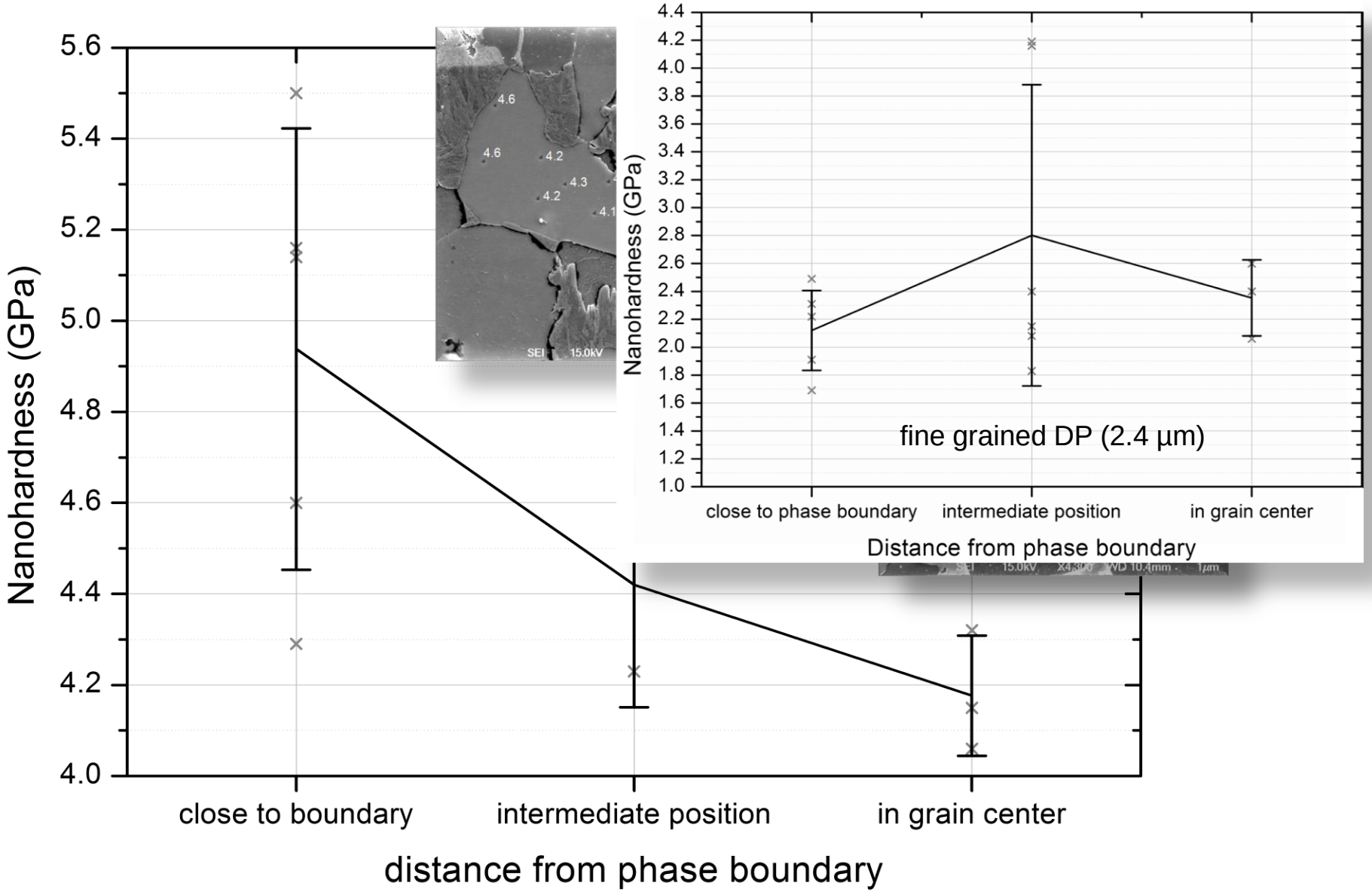
DP steel: effect of GNDs on local hardness?

Coarse grained DP (12.4 µm), Berkovich 50 nm, 500 µN



Calcagnotto et al. Mater. Sc. Engin. A 527 (2010) 2738

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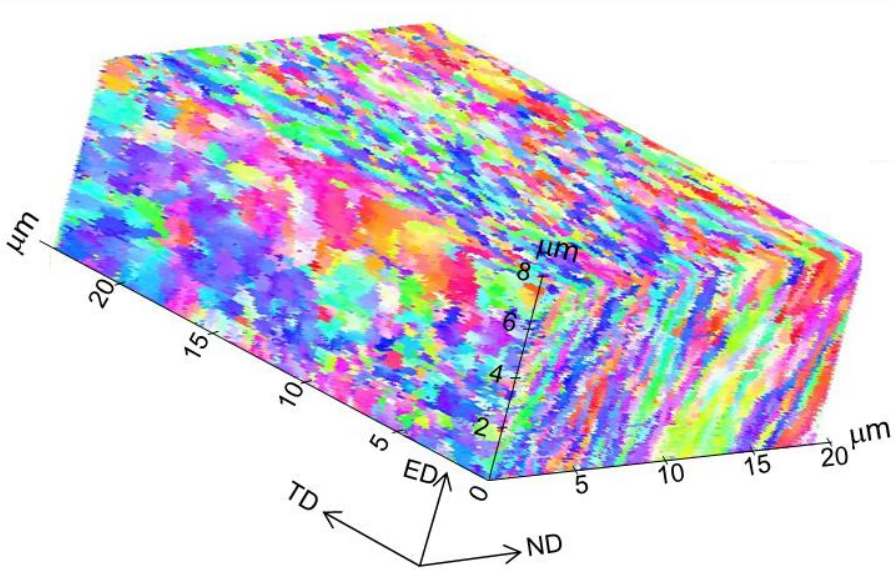
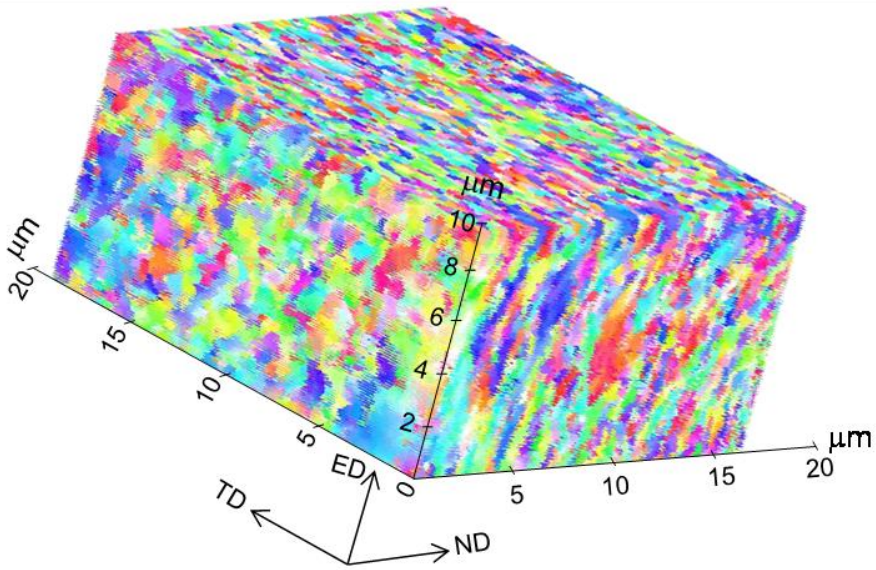
ECAP processing: 3D microstructure and interface analysis

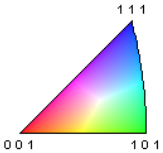
Cu-0.17wt%Zr – as deformed

4 ECAP passes



8 ECAP passes





color: crystal direction parallel to ECAP direction (ED)

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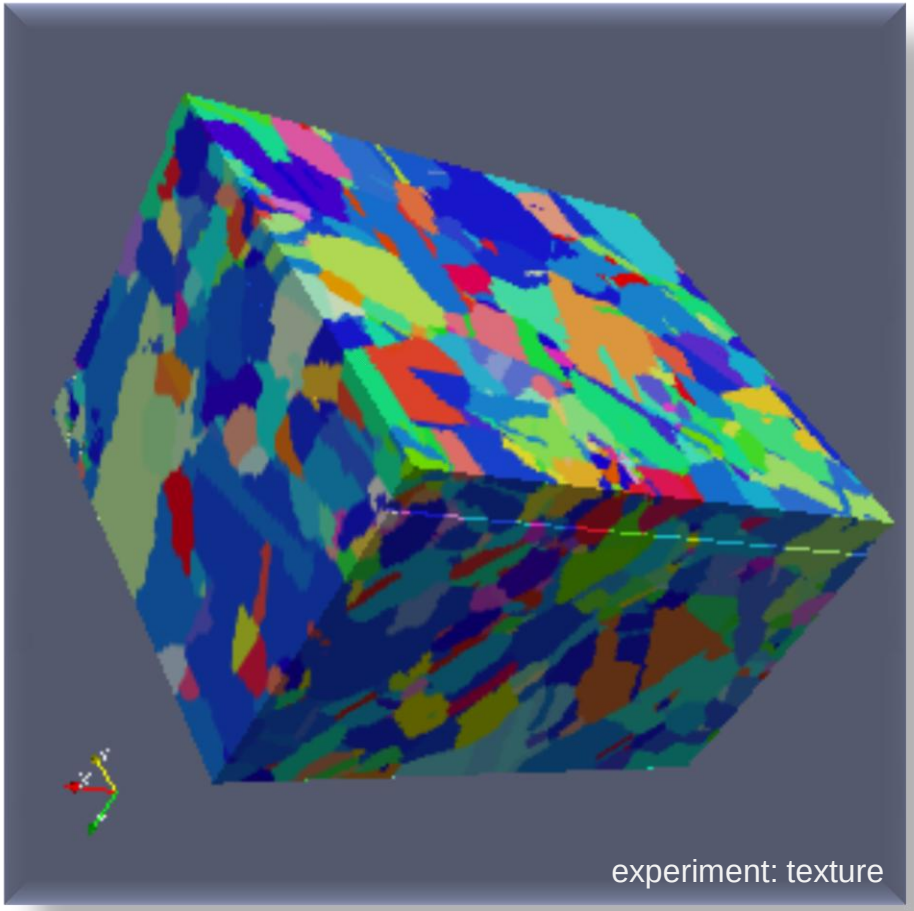
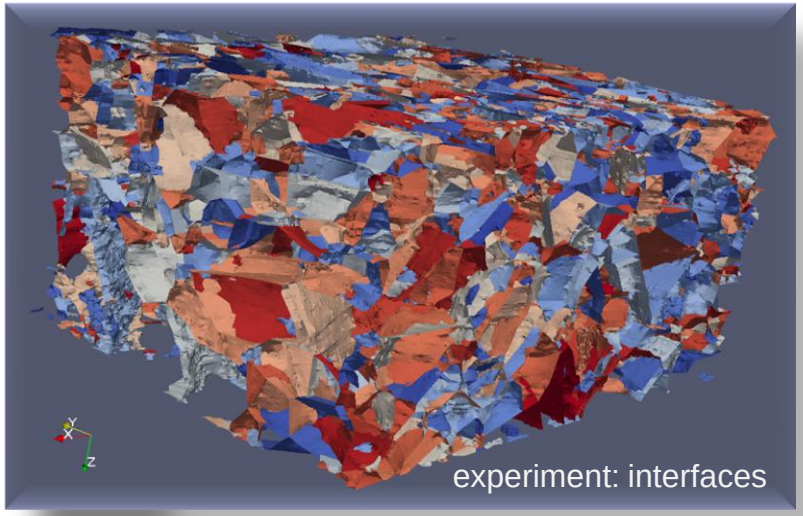
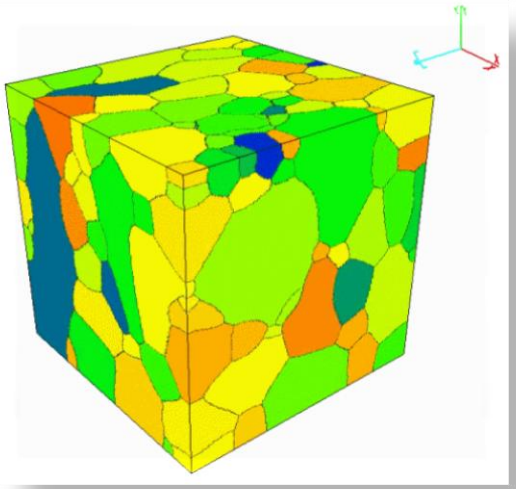
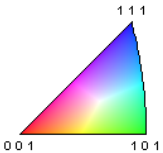
ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – 8 ECAP passes and heat treated at 650°C, 10 min.









color: crystal direction parallel to ECAP direction (ED)

phase field simulation: Melzer, Nestler

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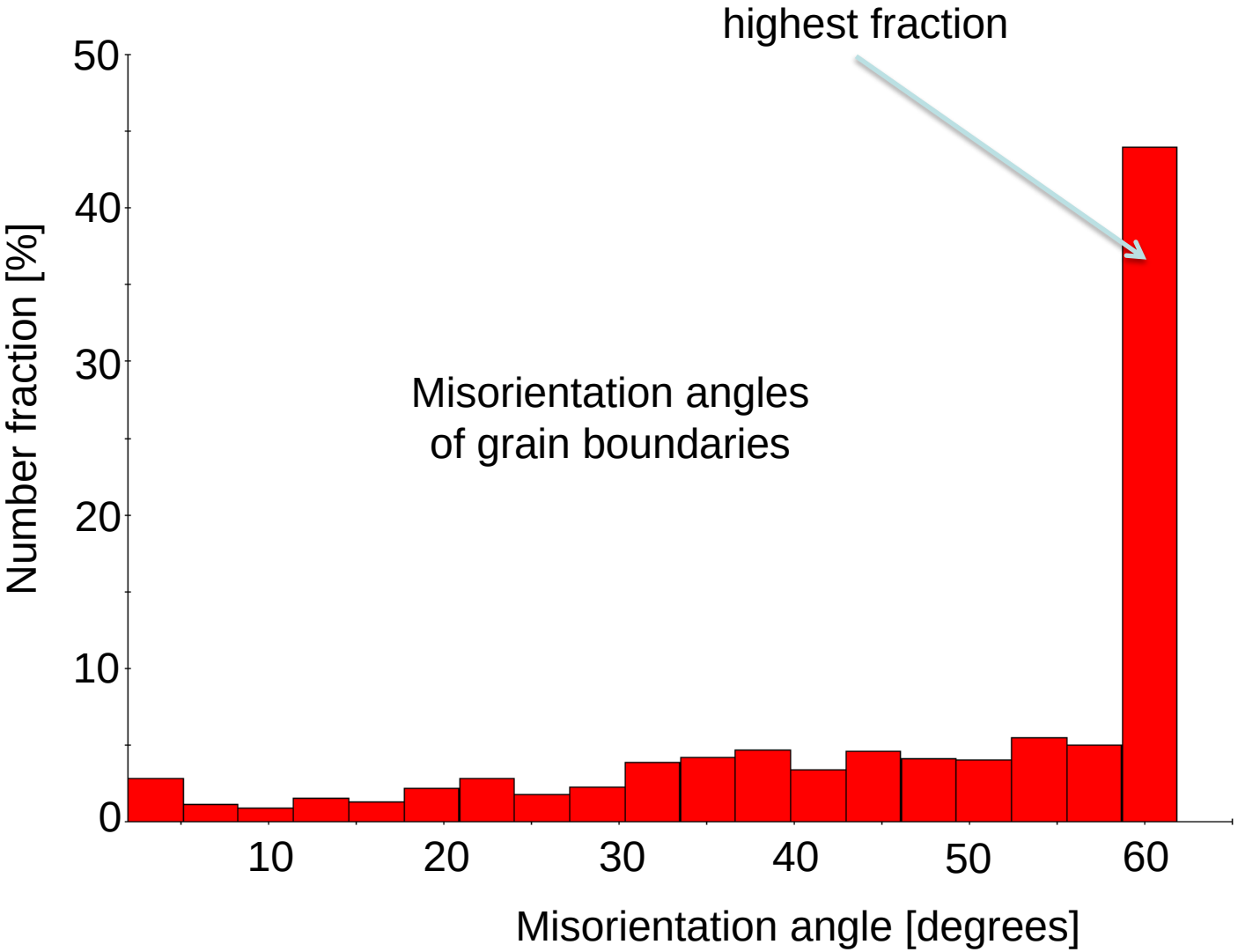
650°C 10 min, Approaches to 8D interface analysis

Cu-0.17wt%Zr – 8 ECAP passes and heat treated at 650°C, 10 min.



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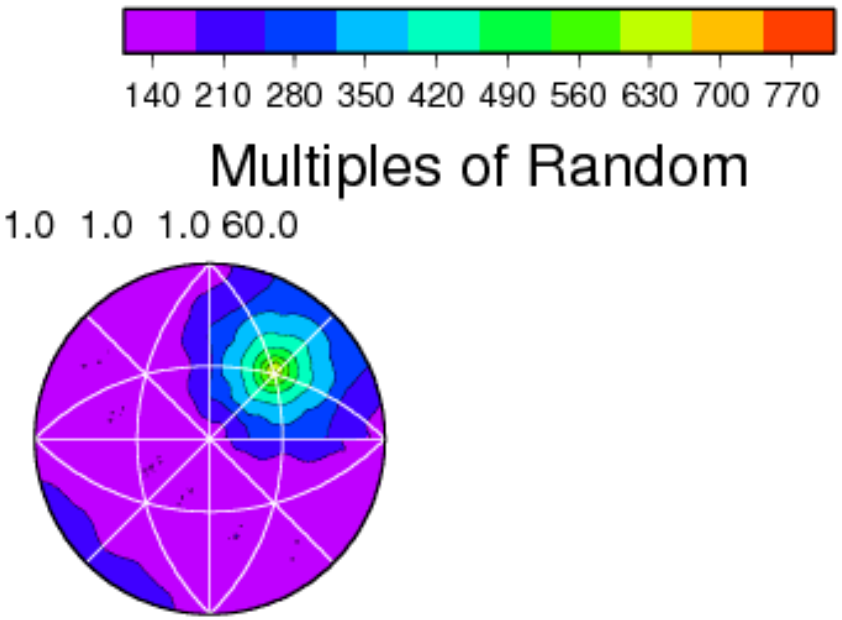
650°C 10 min, interface stereology

Cu-0.17wt%Zr – 8 ECAP passes and heat treated at 650°C, 10 min.

60° [111]



Max. 818.70

Resolution: 8.18°

CLS method: Rollett

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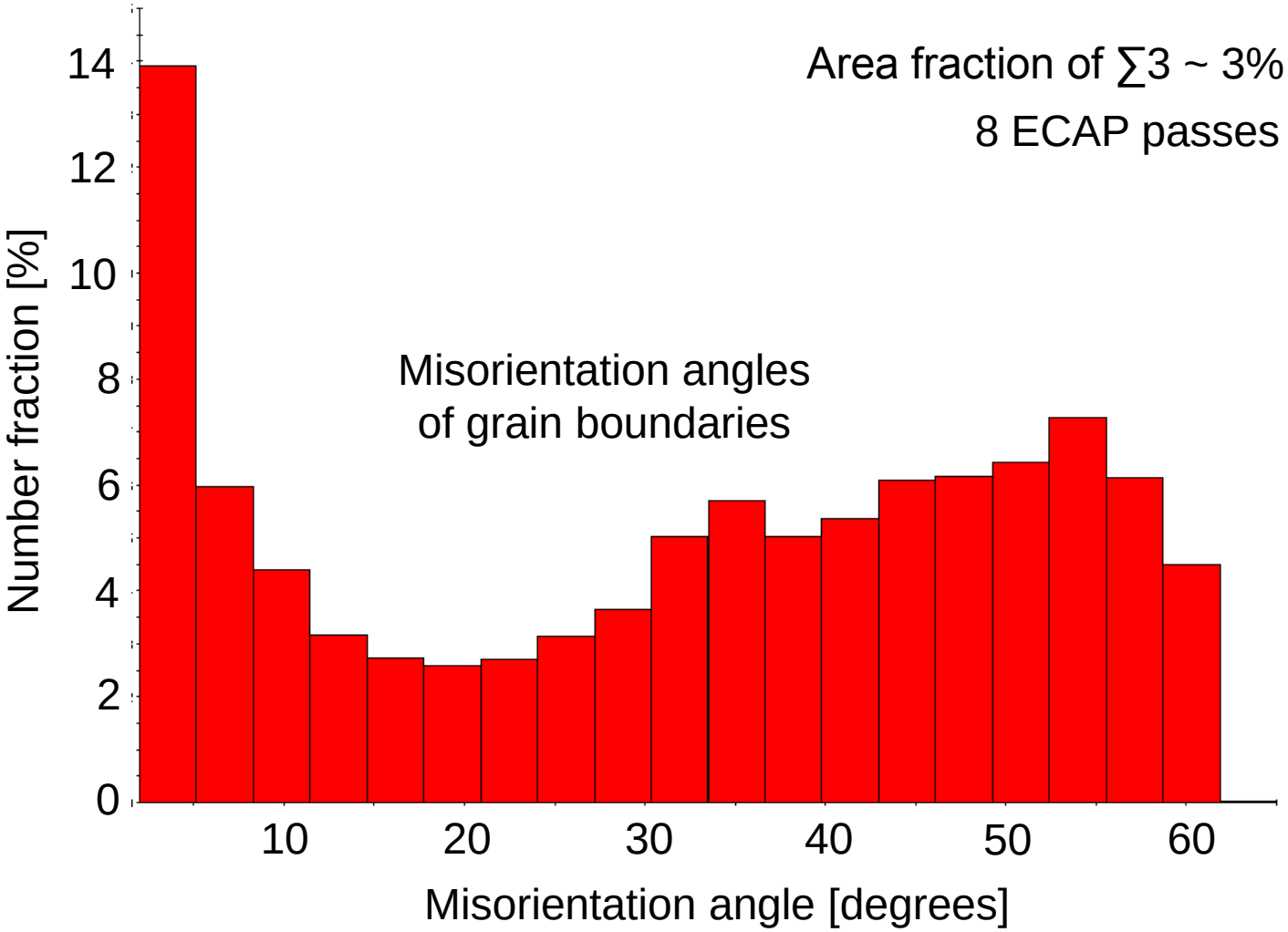
ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – as deformed after 8 passes



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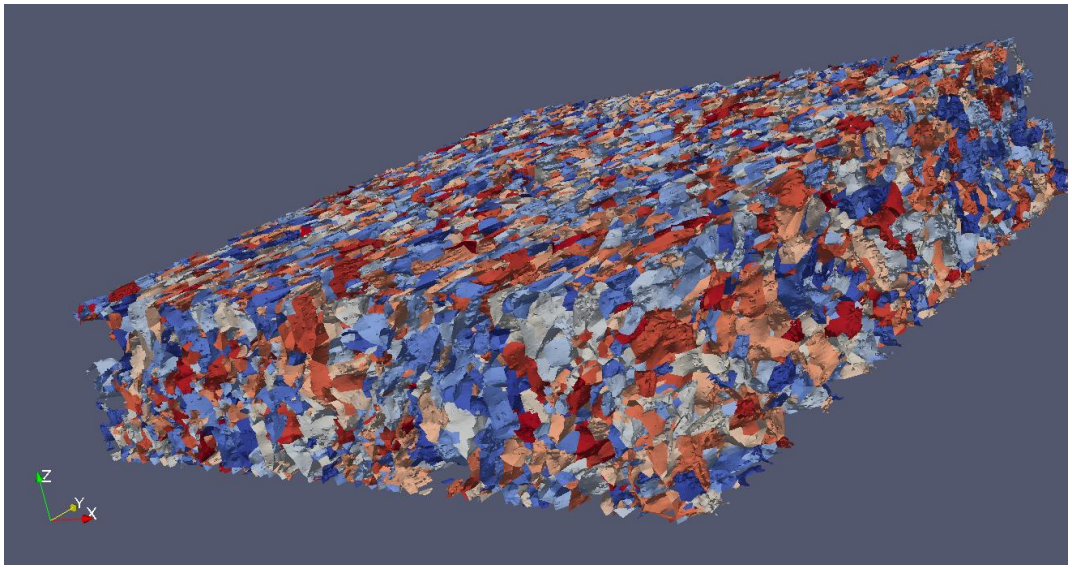
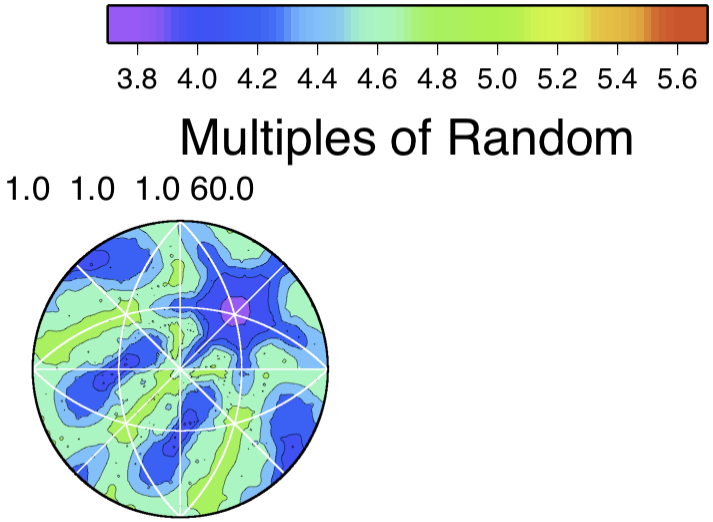
ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – as deformed after 8 passes

Resolution: 8.18°





Max. 5

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Overview

- **Motivation and Features**
- **Methods**

  Experimental set-up

  Data analysis (GND; interface textures;

  simulation: CPFEM, phase field, MC, CA)
- **Examples**

  Indentation: GND

  DP steels: GND

  ECAP processing: interface textures
- **Conclusions**

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Conclusions

- A multi-dimensional microstructure vector is obtained at each 3D spatial position
  - phase, orientation, defect density, elemental composition
- Spatial resolution: 50 x 50 x 50 nm^3
- Observable volume: ≈ 50 x 50 x 50 μm^3
- Angular resolution: 0.5° (precision of tilt)
- Time per cut: 15 … 60 min /cycle
- Examples: Size effect, interface crystallography, interface strengthening